

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED / ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		ATTORNEY'S DOCKET NUMBER P65124USO 097508094
INTERNATIONAL APPLICATION NO. PCT/FR98/01881	INTERNATIONAL FILING DATE 3 September 1998	PRIORITY DATE CLAIMED 16 September 1997
TITLE OF INVENTION METHOD AND DEVICE FOR TREATING A HEAVILY LOADED LIQUID EFFLUENT IN PARTICULAR WITH NITROGEN AND WITH PHOSPHORUS		
APPLICANT(S) FOR DO/EO/US Claude JOLY		

Applicant herein submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information.

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for Internatl. Preliminary Examination was made by the 19th month from earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☒ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☐ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ A translation of the annexes to the Internatl. Preliminary Examination report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
☐ A SECOND or SUBSEQUENT preliminary amendment.
14. ☐ A substitute specification.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information:

International Search Report -- EPO
 First Page of Published Application
 International Preliminary Examination Report (IPER) --
 with Annexes in French

US APPLICATION NO. (If known, see 37 CFR 1.492) <div style="font-size: 1.5em; font-weight: bold;">097308094</div>		INTERNATIONAL APPLICATION NO. PCT/FR98/01881		ATTORNEY'S DOCKET NUMBER P65124US0	
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17. <input checked="" type="checkbox"/> The following fees are submitted: Basic National Fee (37 CFR 1.492(a)(1)-(5)): Internatl. prelim. examination fee paid to USPTO (37 CFR 1.492 (a) (1)) .. \$670.00 No international preliminary examination fee paid to USPTO (37 CFR 1.492 (a) (2)) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) .. \$760.00 Neither international preliminary examination fee (37 CFR 1.492 (a) (3)) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO) \$970.00 International preliminary examination fee paid to USPTO (37 CFR 1.492 (a) (4)) and all claims satisfied provisions of PCT Article 33(2)-(4) \$96.00 Search Report prepared by the EPO or JPO (37 CFR 1.492 (a) (5)) \$840.00 <div style="text-align: right;">ENTER APPROPRIATE BASIC FEE AMOUNT =</div>				CALCULATIONS		PTO USE ONLY	
				\$ 840.00			

Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$ 130.00			
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Claims	Number Filed	Number Extra	Rate		
Total Claims	12 - 20 =	-0-	x \$18.00	\$	
Independent Claims	1 - 3 =	-0-	x \$78.00	\$	
Multiple Dependent Claim(s) (if applicable)			+ \$260.00	\$	
TOTAL OF ABOVE CALCULATIONS =				\$ 970.00	

Reduction by 1/2 for filing by small entity , if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				\$			
SUBTOTAL =				\$ 970.00			

Processing fee of \$130 for furnishing the English translation later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f))				\$ 130.00			
TOTAL NATIONAL FEE =				\$ 1100.00			

Fee of \$40.00 for recording the enclosed assignment (37 CFR 1.21(h)). Assignment must be accompanied by appropriate cover sheet (37 CFR 3.28, 3.31).				\$			
TOTAL FEES ENCLOSED =				\$ 1100.00			

				Amt. to be refunded:		\$	
				Amt. charged:		\$	

a. ☒ A check in the amount of \$ 1100.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. 06-1358 in the amount of \$ to cover the above fees.
A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge my account any additional fees set forth in §1.492 during the pendency of this application, or credit any overpayment to Deposit Account No. 06-1358. A duplicate copy of this sheet is enclosed.

SEND ALL CORRESPONDENCE TO:
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 Washington, DC 20004
 202-638-6666
CUSTOMER NUMBER: 00136

By

D. Douglas Price
Reg. No. 24,514

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s): Claude JOLY

Serial No.: 09/508,094

Filed: March 16, 2000

For: METHOD AND DEVICE FOR TREATING A HEAVILY LOADED
LIQUID EFFLUENT IN PARTICULAR WITH NITROGEN AND
WITH PHOSPHORUS

PRELIMINARY AMENDMENT TO LESSEN FEES

Assistant Commissioner of Patents
Washington, D.C. 20231

Sir:

Prior to initial examination, please amend the above-
identified application as follows:

IN THE CLAIMS

Claim 7, line 1, delete "Claims 1 to 5",

insert --claim 1--;

Claim 8, line 1, delete "Claims 1 to 7",

insert --claim 1--.

REMARKS

The foregoing Preliminary Amendment is requested in order to
delete the multiple dependent claims and avoid paying the multiple
dependent claims fee.

Early action on the merits is respectfully requested.

Respectfully submitted,

JACOBSON, PRICE, HOLMAN & STERN, PLLC

By

D. Douglas Price
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Date: June 2, 2000
Atty. Docket: P65124US0
DDP/cmf

Title: Method and device for treating a liquid effluent heavily loaded especially with nitrogen and phosphorus

The present invention relates to a method for treating a liquid effluent heavily loaded especially with nitrogen and phosphorus, and also a device for implementing this method.

Liquid effluents heavily loaded especially with nitrogen and phosphorus, whether of urban, agricultural or industrial origin, pose a serious problem especially in the field of the environment. All the developing controversy concerning the level of nitrate present in ground water must be borne in mind.

National and/or international legislations tend increasingly to prohibit any disposal of liquid effluents, the amount of nitrogenized material of which is greater than a threshold which is tending to fall as a result of action by various pressure groups. Likewise as regards phosphorus, the legislation limits the quantities tolerated for materials which are to be spread as manure.

Furthermore, a benefit from the above-mentioned liquid effluents is often obtained by spreading them as manure, the nauseating odour which results being a source of conflict with the surrounding neighbourhood.

Amongst the liquid effluents heavily loaded especially with nitrogen and/or with phosphorus, primarily pig slurry should be mentioned, and also liquid sludge created by purification stations.

Indeed, a certain number of methods and/or devices have already been proposed for reducing the nuisance caused by the spreading of such liquid effluents, but none of these is entirely satisfactory, especially with respect to the regulations which are currently envisaged.

Thus, one of the aims of the present invention is to provide a method for treating a liquid effluent heavily loaded especially with nitrogen and with phosphorus, which allows the nitrogenized charge to be reduced at least to the legally acceptable threshold.

Another aim of the present invention is to provide a method of this nature which allows such liquid effluents to be deodorized.

An additional aim of the invention is to provide such a method which is simple to implement at low cost.

These aims, and also others which will later become apparent, are achieved by a method for treating a liquid effluent heavily loaded especially with nitrogen and with phosphorus which is, in accordance with the present invention, characterised in that it comprises the following stages:

- a) the addition of a basic reagent to this liquid effluent to obtain a pH in the range from 8.5 to 13; and
- b) pulverisation of the basified liquid effluent derived from stage a) in a stream of air.

Advantageously, the basic reagent added to stage a) is unslaked or slaked lime in the form of powder, paste or liquid. This reagent may have a lime concentration $[\text{Ca}(\text{OH})_2]$ up to 1000 g/litre of reagent.

Preferably, stage b) is repeated a certain number of times for the same basified effluent: the passage number is in the range from 1 to 50.

Advantageously, at the start of stage b) an anti-foam catalyst is added, the quantity of which varies from 0 to 1 litres for cubic metre of liquid effluent which is to be treated.

According to a preferred form of embodiment, the method in accordance with the present invention may comprise a third stage or stage c) of sifting of the liquid effluent derived from stage b).

As previously indicated, the present invention likewise concerns a device for implementing the above method, which is of low cost. This device comprises:

- a mixing reactor for contacting the liquid effluent with the basic reagent, provided with an intake for this effluent and another intake for the basic reagent;
- an ammonia-extracting reactor, connected to the mixing reactor, and
- a tank for storing the treated liquid effluent derived from the ammonium-extracting reactor.

Advantageously, the mixing reactor comprises a device for measuring the pH of the medium connected to a means situated on the intake of basic reagent to automatically regulate the quantity thereof which is added.

Preferably, the ammonia-extracting reactor or degassing reactor comprises a lower part collecting especially the basified liquid effluent and an upper part in which is situated a pulverisation rack provided with nozzles, connected to the lower part of the reactor and comprising a feed pump, openings being provided between the two parts so that the exterior air can enter, an air exhaust fan being connected to this upper part. The nozzles of the pulverisation rack are, for example, of the cyclone type.

Advantageously, the upper part of the degassing reactor is connected to a devesiculer.

The following description, which is in no way restrictive in character, is to be read with reference to the single attached figure which is a diagrammatic vertical section of a part of a device for implementing the method according to the present invention.

In accordance with the invention, a method for treating a liquid effluent heavily loaded especially with nitrogen and/or with phosphorus comprises a first stage or stage a) of mixing a basic reagent with the liquid effluent which is to be treated. The pH of this effluent is thus brought to a value in the range from 8.5 to 13.

The basic reagent is essentially composed of unslaked or slaked lime, present in the form of powder, liquid or paste and being able to have a concentration reaching 1000 g of equivalent $\text{Ca}(\text{OH})_2$ per litre of reagent.

This addition, by raising the pH, allows one to transform the nitrogenized compounds which are present in the liquid effluent in the form of ammonium ions, symbolised by the name N-NH_4^+ into gaseous ammonia (NH_3) dissolved in the liquid effluent.

Furthermore, the presence of lime leads to the precipitation in the form of calcic compounds the phosphatic compounds present in this effluent in the form of orthophosphates. Likewise the sulphurated compounds which are emitted in the form of a bad-smelling gas, sulphur dioxide (H_2S), are transformed into calcium sulphate, an inert product.

In a second stage or stage b) for extraction of ammonia, the liquid effluent derived from the preceding stage is transferred and pulverized in an air stream: the gaseous ammonia (NH_3) is entrained by the air. The transformation of the ammonium ions into ammonia is all the faster since the pH is in the range from 9.5 to 12.5. The temperature of 70°C maximum also increases the degassing.

At the start of this stage b), an anti-foam catalyst can be added, the quantity of which varies from 0 to 1 l/m³ of liquid effluent which is to be treated.

The method in accordance with the invention possibly comprises a third stage, or stage c) of sifting, allowing the separation of the particles in suspension after decanting, before storing the liquid effluent which has been treated.

The present invention likewise concerns a device for implementing the method described above.

This device comprises, firstly, a mixing reactor, not shown in the figure, in which the first stage is carried out. This reactor comprises an intake for the liquid effluent and an intake for the basic reagent. It is provided with a stirring means for mixing as intimately as possible the liquid effluent to be treated with the basic reagent.

This mixing reactor may also comprise means for the regulation of flow arranged on the intake for the basic reagent which are connected to a device for measuring the pH of the medium in reaction. This basic reagent, which is essentially made up of unslaked or slaked lime, is incorporated by sequences which are timed and repeated successively as many times as necessary until the desired pH is obtained : this can be realized automatically. Each incorporation sequence comprises four phases:

- measurement of the initial pH of the loaded liquid effluent,
- injection of dilution liquid of the basic reagent during a given time, for example 30 minutes, which liquid may be water, this operation being able to be carried out in masked time;

- the incorporation of the basic reagent diluted in the effluent to be treated during a given time, for example during successive periods each of 20 seconds,
- mixing of the effluent and of the basic reagent especially by stirring.

The duration of a sequence lasts from 30 minutes to 3 hours, in general.

The device in accordance with the present invention comprises, secondly, a degassing reactor designated as a whole by reference number 1 and connected to the mixing reactor.

This degassing reactor 1 is composed of two parts:

- a lower part 2 intended to receive the mixture derived from the mixing reactor. This lower part may, according to a preferred form of embodiment, be composed of several tanks 2a, 2b, 2c, each connected to the same evacuation pipe 3 towards a pulverisation rack 4;
- an upper part 5 comprising the pulverisation rack 4 which is provided with nozzles 6, for example of the cyclone type, which is connected to the lower part 2 of the reactor and is provided at this level with a pump 7: this pump 7 draws, in the pulverisation rack 4, the mixture present in the lower part 2. Openings 8 are arranged between the lower part 2 and upper part 5 to permit an intake of exterior air, an exhaust fan, not shown, being connected to this upper part: thus, an air stream is formed. As regards the exhaust fan, it causes a circulation of the air which is charged with ammonia whilst crossing the sheet of droplets of the mixture derived from the pulverisation rack 4.

This rack 4 can also comprise an intake for an anti-foam product: the latter has the aim of maintaining a regular fluidity to the basified liquid effluent.

The product derived from the nozzles is recovered in the upper part and recycled towards the pulverisation rack: the cycle number can reach 50, to obtain a given level of extraction of fixed ammoniacal nitrogen.

The treated liquid effluent derived from the degassing stage b) is conveyed towards storage tanks, possibly after sifting to eliminate especially the phosphatic compounds.

As regards the air drawn in the upper part, it is drawn out from the degassing reactor and is conveyed in a devesiculer 9 so as to relieve it of the droplets of liquid with which it has become loaded.

This air loaded with ammonia is returned into the atmosphere if the concentration is lower than the authorized ejection standards, or treated in a washing tower or by any other means allowing the ammonia to be collected or eliminated.

When the air loaded with ammonia is treated, the air which is thus purified can be recycled towards the openings arranged between the lower part and upper part of the degassing reactor.

To remove 80% of the ammoniacal nitrogen contained in a pig slurry, the device in accordance with the present invention allows approximately 1 to 4 m³ of slurry to be treated per hour; the flow of air must be in the range from 6,000 to 15,000 m³/h.

To remove 60% of the ammoniacal nitrogen, this device allows approximately 2 to 8 m³/h pig slurry to be treated, with the same air flow as above.

To remove 40% of the ammoniacal nitrogen, this device allows approximately 3 to 10 m³/h pig slurry to be treated, the air flow being the same.

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Claims:

1. A method for treating a liquid effluent heavily loaded especially with nitrogen and with phosphorus, characterised in that it comprises the following stages:
 - a) addition of a basic reagent to this liquid effluent to obtain a pH in the range from 8.5 to 13; and
 - b) pulverisation of the basified liquid effluent derived from stage a) in a stream of air.
2. The method according to Claim 1, characterised in that the basic reagent added to stage a) is unslaked or slaked lime in the form of powder, paste or liquid.
3. The method according to Claim 2, characterised in that the concentration of lime $[\text{Ca}(\text{OH})_2]$ is a maximum of 1,000 g/litre of reagent.
4. The method according to Claim 3, characterised in that the stage b) is repeated a certain number of times for the same basified effluent.
5. The method according to Claim 4, characterised in that the passage number is in the range from 1 to 50.
6. The method according to Claim 1, characterised in that at the start of stage b) an anti-foam catalyst is added, the quantity of which varies from 0 to 1 l/m³ of liquid effluent which is to be treated.
7. The method according to Claims 1 to 5, characterised in that it likewise comprises a stage c) for sifting the liquid effluent derived from stage b).
8. A device for implementing the method according to Claims 1 to 7, characterised in that it comprises
 - a mixing reactor for bringing the liquid effluent into contact with the basic reagent, provided with an intake for this effluent and another intake for the basic reagent;
 - an ammonia-extracting reactor (1), connected to the mixing reactor, and
 - a tank for storing the treated liquid effluent derived from the ammonia-extracting reactor(1).
9. The device according to Claim 8, characterised in that the mixing reactor comprises a device for measuring the pH of the medium connected to a means situated on the intake for the basic reagent for regulating automatically the added quantity thereof.
10. The device according to Claim 9, characterised in that the ammonia-extracting reactor (1) or degassing reactor comprises a lower part (2) collecting in particular the basified liquid effluent and an upper part (5) in which there is situated a pulverisation rack (4) provided with nozzles (6), connected at the lower part (2) to said reactor (1) and

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comprising a feed pump (7), openings (8) being arranged between the two parts to allow exterior air to enter, an exhaust air fan being connected to said upper part (5).

11. The device according to Claim 10, characterised in that the pulverisation rack (4) comprises nozzles (6) of the cyclone type.

12. The device according to Claim 10, characterised in that the upper part (5) of the degassing reactor (1) is connected to a devesiculer (9).

13. The device according to Claim 12, characterised in that it comprises a washing tower connected to the devesiculer or any other means allowing the ammonia to be collected or eliminated.

002090-4000000

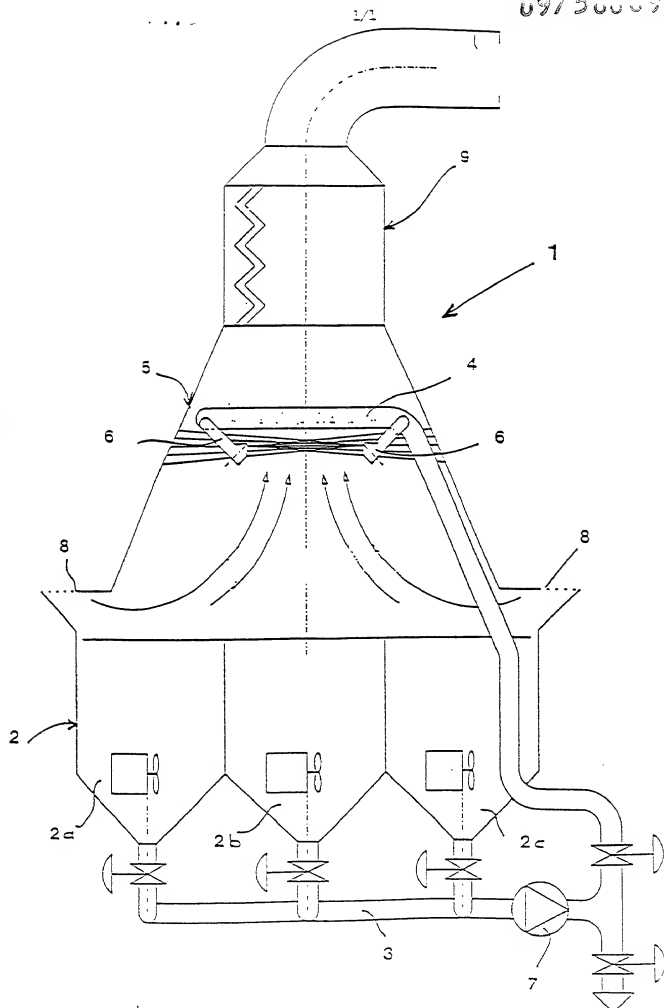


FIG. 1

**DECLARATION
AND POWER OF ATTORNEY
U.S.A.**

FOR ATTORNEYS' USE ONLY

JUN 2000

ATTORNEYS' DOCKET NO.
P65124US0

ALL PATENTS, INCLUDING DESIGN
FOR APPLICATION BASED ON PCT, PARIS CONVENTION,
NON PRIORITY, OR PROVISIONAL APPLICATIONS

As a below named inventor, I declare that my residence, post office address and citizenship are stated below next to my name, the information given herein is true, that I believe that I am the original, first and sole inventor (if only one name is listed at 201 below), or a first and joint inventor (if plural inventors are named below at 201-203, or on additional sheets attached hereto) of the subject matter which is claimed and for which patent is sought on the invention entitled:

**METHOD AND DEVICE FOR TREATING A HEAVILY LOADED LIQUID EFFLUENT
IN PARTICULAR WITH NITROGEN AND WITH PHOSPHORUS**

which is described and claimed in: ☒ PCT International Application No. PCT/FR98/01881 filed September 3, 1998

☐ the attached specification

☐ the specification in application Serial No. _____

filed _____

(if applicable) and amended on _____

I hereby state that I have reviewed and understand the contents of the above-identified specifications, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 (a)-(d) of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Claimed

97/11478
(Number)

France
(Country)

16 September 1997 ☒ ☐
(Day/Month/Year Filed) Yes No

(Number)

(Country)

(Day/Month/Year Filed) ☐ ☐

(Number)

(Country)

(Day/Month/Year Filed) ☐ ☐

(Number)

(Country)

(Day/Month/Year Filed) ☐ ☐

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below:

Application No. _____ Filing Date _____ Application No. _____ Filing Date _____

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §121, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

(Application Serial No.)

(Filing Date)

(Status: patented, pending, abandoned)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorneys (Registration No.) to prosecute this application, receive and act on instructions from my agent, and transact all business in the Patent and Trademark Office connected therewith. HARVEY B. JACOBSON, JR. (20,651); D. DOUGLAS PRICE (24,514); JOHN CLARKE HOLMAN (22,769); MARVIN R. STERN (20,640); MICHAEL R. SLOBASKY (26,421); JONATHAN L. SCHERER (29,854); STANFORD W. BERMAN (17,809); IRWIN M. AISENBERG (19,007); WILLIAM E. PLAYER (31,409)

SEND CORRESPONDENCE TO

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JACOBSON, PRICE, HOLMAN & STERN
PROFESSIONAL LIMITED LIABILITY COMPANY

*Inventor(s) name must include at least one unabbreviated first or middle name.

201	FULL NAME* OF INVENTOR	FAMILY NAME <u>JOLY</u>	GIVEN NAME <u>Claude</u>	MIDDLE NAME
	RESIDENCE & CITIZENSHIP	CITY <u>Meylan</u>	STATE OR FOREIGN COUNTRY <u>France</u>	COUNTRY OF CITIZENSHIP <u>France</u>
	POST OFFICE ADDRESS	POST OFFICE ADDRESS <u>38, chemin d'Avat</u>	CITY <u>F-38240 Meylan</u>	STATE OR COUNTRY <u>France</u> ZIP CODE
202	FULL NAME* OF INVENTOR	FAMILY NAME	GIVEN NAME	MIDDLE NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE OR COUNTRY ZIP CODE
203	FULL NAME* OF INVENTOR	FAMILY NAME	GIVEN NAME	MIDDLE NAME
	RESIDENCE & CITIZENSHIP	CITY	STATE OR FOREIGN COUNTRY	COUNTRY OF CITIZENSHIP
	POST OFFICE ADDRESS	POST OFFICE ADDRESS	CITY	STATE OR COUNTRY ZIP CODE

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

SIGNATURE OF INVENTOR 201*	SIGNATURE OF INVENTOR 202*	SIGNATURE OF INVENTOR 203*
<u>Claude JOLY</u>		
DATE <u>20/03/2000</u>	DATE	DATE

☐ Additional inventors are named on separately numbered sheets attached hereto

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